

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7:		(11) International Publication Number: WO 00/09620
C09J 4/06, G11B 7/24, C08F 290/14, 290/06, C08G 18/67	A1	(43) International Publication Date: 24 February 2000 (24.02.00)
(21) International Application Number: PCT/NL (22) International Filing Date: 11 August 1999 (CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC,
(30) Priority Data: 10230469 17 August 1998 (17.08.98)		Published With international search report.
(71) Applicants (for all designated States except US): D [NL/NL]; Het Overloon 1, NL-6411 TE Heerl JSR CORPORATION [JP/JP]; 2-11-24, Tsukiji, Tokyo 104-8410 (JP). JAPAN FINE COATIN LTD. [JP/JP]; 2-11-24, Tsukiji, Chuo-ku, Tokyo (JP).	en (N) Chuo-l GS C	.). u, D.,
(72) Inventors; and (75) Inventors/Applicants (for US only): TAKAHASH hiko [JP/BE]; Avenue de Calabre 36, Boîte 2 Woluwe Saint Lambert (BE). TAKEHANA [JP/JP]; 2-15-2-401 Umezono, Tsukuba, Iba (JP). TAKASE, Hideaki [JP/CA]; 82 Ontario SSI Kingston, Ontario K7L 5M2 (CA). UKACHI (JP/JP]; 5-22-9, Kamiya, Ushiku 300-12 (JP). (74) Agent: DEN HARTOG, Jeroen, Hendrikus, Joseph; C reau DSM, P.O. Box 9, NL-6160 MA Geleen (N	, B-12 , Yuidaraki 3 reet #4 , Taka	00 hi 05 01, shi
(54) Title: PHOTOCURABLE RESIN COMPOSITION	HTIW	A LOW CHLORINE CONTENT

(57) Abstract

A photocurable resin composition comprising (A) an urethane (meth)acrylate prepared by the reaction of a polyol compound, polyisocyanate compound, and hydroxyl group—containing (meth)acrylate compound, (B) a (meth)acrylate compound having at least one (meth)acryloyl group in the molecule, and (C) a photo-initiator, and having a chlorine content of not more than 0.001 wt %. The invention also relates to a process for preparing the photocurable resin composition and an information recording disk fabricated using the photocurable resin composition as an adhesive.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
ΑU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
ВА	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	ТJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	1E	Ireland	MN	Mongolia	UA	Ukraine
BR -	Brazil	1L	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	zw	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ.	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
Cυ	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

PHOTOCURABLE RESIN COMPOSITION WITH A LOW CHLORINE CONTENT

Field of the Invention

5

25

30

The present invention relates to

10 photocurable resin compositions having a low chlorine content. In particular, the present invention relates to a photocurable resin compositions suitable for use with information disks, including in particular, disks having multiple elements comprising information (or the ability to receive and retain information), preferably, this will include optical disks (i.e., those wherein the information, preferably digital information, is recorded and/or retrieved using optics) for example, digital video disks or digital versatile disks,

20 commonly referred to as a DVD's.

Background of the Invention

Recent progress of information technologies including computer devices, computer software, and communication technologies has allowed a high speed conveyance of mass information. Along with this advancement, recording media which can record much more information at high density have been desired and the development of such recording media is ongoing. An example of such high-density recording media is a DVD which has been developed as a recording media for wide use in the next generation. The DVD differs from

PCT/NL99/00511

- 2 -

WO 00/09620

conventional CDs (compact disks) in that the DVD is manufactured by adhering two sheets of disks.

Therefore, an adhesive for adhering the two sheets of disks is required and use of hot-melt, heat-curable, anaerobic curable, or similar adhesives has been attempted.

However, hot-melt adhesives have insufficient heat stability and weatherability. Because these adhesives soften at a high temperature, the decreased adhesive strength causes the adhered disks to be separated and deformed. In addition, since transparency of the adhesive is insufficient, it is difficult to use these adhesives for the DVD comprising two recording films of which one is translucent.

15 A problem with heat-curable adhesives is that heat during curing causes the substrate for forming a disk to deform and the curing time is long.

On the other hand, anaerobic curable adhesives require a certain period of time for curing, thereby resulting 20 in reduced productivity.

Photocurable adhesives have been proposed to solve these problems. For example, UV curable adhesives comprising a urethane acrylate as a major component have been disclosed in Japanese Patent

25 Applications Laid-open No. 142545/1986 and No. 89462/1994. However, optical disks manufactured using these conventional photocurable adhesives exhibit only insufficient moisture resistance and heat resistance. Information recorded in such disks may be lost over time under high temperature and high humidity

- 3 -

conditions.

10

15

20

Summary of the Invention

The present invention provides a

5 photocurable resin composition useful as an adhesive
for fabricating information disks exhibiting superior
heat resistance and moisture resistance.

The present inventors have undertaken extensive studies and found that a photocurable resin composition with a reduced chlorine content, when used as an adhesive for information disks, induces almost no deterioration of metallic films on substrate surfaces for optical disks and can produce information recording disks with excellent moisture resistance and heat resistance.

Specifically, an object of the present invention is to provide a photocurable resin composition comprising (A) an urethane (meth)acrylate, (B) a (meth)acrylate compound having at least one (meth)acryloyl group in the molecule, and (C) a photoinitiator, wherein the content of chlorine in the composition is not more than 0.001 wt%.

Another object of the present invention is to provide a process for preparing the photocurable

25 resin composition according to claim 1, comprising a step of washing the components (A), (B), and (C) either prior, during or after blending two or more of the components (A), (B), and (C).

Still another object of the present

30 invention is to provide an information disk comprising

- 4 -

two elements adhered to each other using the above photocurable resin composition.

Preferred Embodiments of the Invention

The urethane (meth) acrylate used as the component (A) is obtained by reacting a polyol compound, a polyisocyanate compound, and a hydroxyl group-containing (meth) acrylate.

invention, a polyether polyol, polyester polyol, polycarbonate polyol, polycaprolactone polyol, aliphatic hydrocarbon containing two or more hydroxyl groups in the molecule, alicyclic hydrocarbon containing two or more hydroxyl groups in the molecule, unsaturated hydrocarbon containing two or more hydroxyl groups in the molecule, and the like can be given.

These polyol compounds may be used either independently or in combinations of two or more.

The above polyether polyol includes 20 aliphatic polyether polyols, alicyclic polyether polyols, and aromatic polyether polyols.

Examples of the aliphatic polyether polyols include polyhydric alcohols such as polyethylene glycol, polypropylene glycol, polytetramethylene glycol, polyhexamethylene glycol, polyhexamethylene glycol, polyhexamethylene glycol, pentaerythritol, dipentaerythritol, trimethylolpropane, and alkylene oxide addition polyols such as ethylene oxide addition triol of trimethylolpropane, propylene oxide addition triol of trimethylolpropane, ethylene oxide and

- 5 -

propylene oxide addition triol of trimethylolpropane, ethylene oxide addition tetraol of pentaerythritol, and ethylene oxide addition hexaol of dipentaerythritol; polyether polyols produced by ring-opening polymerization of two or more ionic polymerizable cyclic compounds; and the like.

Given as examples of the ionic

polymerizable cyclic compounds are cyclic ethers such
as ethylene oxide, propylene oxide, butene-1-oxide,

10 isobutene oxide, 3,3-bischloromethyl oxetane,
tetrahydrofuran, 2-methyltetrahydrofuran, dioxane,
trioxane, tetraoxane, cyclohexene oxide, styrene oxide,
epichlorohydrin, glycidyl ether, allyl glycidyl ether,
allyl glycidyl carbonate, butadiene monoxide, isoprene

15 monoxide, vinyloxetane, vinyltetrahydrofuran,
vinylcyclohexene oxide, phenyl glycidyl ether, butyl
glycidyl ether, and glycidyl benzoate.

Examples of specific combinations of two or more of the above ionic polymerizable cyclic compounds

20 include combinations of tetrahydrofuran and ethylene oxide, tetrahydrofuran and propylene oxide, tetrahydrofuran and 2-methyltetrahydrofuran, tetrahydrofuran and 3-methyltetrahydrofuran, ethylene oxide and propylene oxide, butene-1-oxide and ethylene

25 oxide, and tetrahydrofuran, butene-1-oxide, and ethylene oxide.

A polyether polyol produced by ring-opening polymerization of one of the above ionic polymerizable cyclic compounds and cyclic imines such as ethyleneimine, cyclic lactic acids such as β -

- 6 -

propyolactone and glycolic acid, or dimethylcyclopolysiloxane can also be used.

Examples of the alicyclic polyether polyols include alkylene oxide addition diol of hydrogenated bisphenol A, alkylene oxide addition diol of hydrogenated bisphenol F, and alkylene oxide addition diol of 1,4-cyclohexanediol.

Examples of the aromatic polyether polyols include alkylene oxide addition diol of bisphenol A, 10 alkylene oxide addition diol of bisphenol F, alkylene oxide addition diol of hydroquinone, alkylene oxide addition diol of naphthohydroquinone, and alkylene oxide addition diol of anthrahydroquinone.

Polyether polyols are commercially 15 available under the following trade names: examples of aliphatic polyether polyols include PTMG650, PTMG1000, PTMG2000 (manufactured by Mitsubishi Chemical Corp.), PPG1000, EXCENOL1020, EXCENOL2020, EXCENOL3020, EXCENOL4020 (manufactured by Asahi Glass Co., Ltd.), 20 PEG1000, Unisafe DC1100, Unisafe DC1800, Unisafe DCB1100, Unisafe DCB1800 (manufactured by Nippon Oil and Fats Co., Ltd.), PPTG1000, PPTG2000, PPTG4000, PTG400, PTG650, PTG2000, PTG3000, PTGL1000, PTGL2000 (manufactured by Hodogaya Chemical Co., Ltd.), Z-3001-25 4, Z-3001-5, PBG2000, PBG2000B (manufactured by Daiichi Kogyo Seiyaku Co., Ltd.), TMP30, PNT4 Glycol, EDA P4, EDA P8 (manufactured by Nippon Nyukazai Co., Ltd.), and

Quadrol (manufactured by Asahi Denka Kogyo K.K.); and examples of aromatic polyether polyols include Uniol

DA400, DA700, DA1000, and DB400 (manufactured by Nippon

Oil and Fats Co., Ltd.).

The above-mentioned polyester polyol can be obtained by reacting a polyhydric alcohol with a polybasic acid. Examples of a polyhydric alcohol

- include ethylene glycol, polyethylene glycol, propylene glycol, polypropylene glycol, tetramethylene glycol, polytetramethylene glycol,
 - 1,4-butanediol, 1,5-pentanediol, 1,6-hexane diol,
 - 1,7-heptanediol, 1,8-octanediol, neopentyl glycol,
- propylene oxide addition compound of trimethylolpropane, ethylene oxide and propylene oxide addition compound of trimethylolpropane, sorbitol, pentaerythritol, dipentaerythritol, and alkylene oxide addition polyols. As examples of polybasic acids,
- phthalic acid, isophthalic acid, terephthalic acid, maleic acid, fumaric acid, adipic acid, sebacic acid, and the like can be given. As commercially available products of these polyester polyols, Kurapol P1010, Kurapol P2010, PMIPA, PKA-A, PKA-A2, PNA-2000
- 25 (manufactured by Kuraray Co., Ltd.), Tonepolyol 0200,
 Tonepolyol 0221, Tonepolyol 0301, Tonepolyol 0310,
 Tonepolyol 2201, Tonepolyol 2221 (manufactured by Union
 Carbide Corp.), and the like can be used.

Moreover, as examples of the above 30 polycarbonate polyols, polycarbonate diols shown by the

- 8 -

formula (1) are given:

$$\begin{array}{c|c}
0 & & \\
HO - (R^{1} - OCO)_{m} - R^{1} - OH
\end{array} (1)$$

wherein R¹ represents an alkylene group having 2-20 carbon atoms, a residual group of (poly)ethylene glycol, a residual group of (poly)propylene glycol, or a residual group of (poly)tetramethylene glycol, and m is an integer from 1 to 30.

Given as specific examples of R¹ are

residual groups of 1,4-butanediol, 1,5-pentanediol,
neopentyl glycol, 1,6-hexanediol, 1,4cyclohexanedimethanol, 1,7-heptanediol, 1,8-octanediol,
1,9-nonanediol, ethylene glycol, diethylene glycol,
triethylene glycol, tetraethylene glycol, propylene
glycol, dipropylene glycol, tripropylene glycol,
tetrapropylene glycol, and the like.

These polycarbonate polyols are commercially available under the trade names, for example, DN-980, DN-981, DN-982, DN-983 (manufactured by Nippon Polyurethane Industry Co., Ltd.), PC-8000 (manufactured by PPG), PNOC1000, PNOC2000, PMC100, PMC2000 (manufactured by Kuraray Co., Ltd.), PLACCEL CD-205, CD-208, CD-210, CD-220, CD-205PL, CD-208PL, CD-210PL, CD-220PL, CD-205HL, CD-210HL, CD-220HL, CD-220HL, CD-221T (manufactured by Daicel Chemical Industries, Ltd.), and the like.

Examples of the above-mentioned

- 9 -

polycaprolactone polyol include polycaprolactone diols obtained by the addition reaction of ϵ -caprolactone to a diol such as ethylene glycol, polyethylene glycol. propylene glycol, polypropylene glycol, tetramethylene 5 glycol, polytetramethylene glycol, 1,2-polybutylene glycol, 1,6-hexanediol, neopentyl glycol, 1,4cyclohexanedimethanol, and 1,4-butanediol. These are commercially available under the trade names, for example, PLACCEL 205, 205AL, 212, 212AL, 220, 220AL (manufactured by Daicel Chemical Industries, Ltd.) and the like.

Examples of the above aliphatic hydrocarbon containing two or more hydroxyl groups in the molecule include ethylene glycol, propylene glycol,

- 15 tetramethylene glycol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,7-heptanediol, 1,8-octanediol, 1,9nonanediol, neopentyl glycol, 2,2-diethyl-1,3propanediol, 3-methyl-1,5-pentanediol, 2-methyl-1,8octanediol, hydrogenated polybutadiene with terminal 20
- hydroxyl groups, glycerol, trimethylolpropane, pentaerythritol, and sorbitol.

10

25

30

Examples of the above alicyclic hydrocarbon having two or more hydroxyl groups in the molecule include 1,4-cyclohexanediol, 1,4-cyclohexanedimethanol, 1,2-bis(hydroxyethyl)cyclohexane, dimethylol compounds

As examples of the above unsaturated hydrocarbon containing two or more hydroxyl groups in the molecule, a polybutadiene with terminal hydroxyl groups, a polyisoprene with terminal hydroxyl groups,

of dicyclopentadiene, and tricyclodecanedimethanol.

- 10 -

and the like can be given.

Furthermore, examples of polyols other than the above-mentioned polyol compounds include $\beta\text{-methyl-}\delta\text{-valerolactone}$ diol, castor oil-modified diol, a polydimethylsiloxane with diols at the terminals, and a polydimethylsiloxane carbitol-modified diol.

The number average molecular weight of these polyol compounds is preferably from 50 to 15000, and more preferably from 100 to 8000.

As the polyisocyanate compound,
diisocyanate compounds are preferable. Examples of
diisocyanate compounds include 2,4-tolylene
diisocyanate, 2,6-tolylene diisocyanate, 1,3-xylylene
diisocyanate, 1,4-xylylene diisocyanate, 1,5-

naphthalene diisocyanate, m-phenylene diisocyanate, pphenylene diisocyanate, 3,3'-dimethyl-4,4'diphenylmethane diisocyanate, 4,4'-diphenylmethane
diisocyanate, 3,3'-dimethylphenylene diisocyanate,
4,4'-biphenylene diisocyanate, 1,6-hexane diisocyanate,

isophorone diisocyanate, 2,2,4-trimethylhexamethylene diisocyanate, bis(2-isocyanate ethyl) fumarate, 6-isopropyl-1,3-phenyl diisocyanate, 4-diphenylpropane diisocyanate, lysine diisocyanate, hydrogenated diphenylmethane diisocyanate, hydrogenated xylylene

diisocyanate, and tetramethylxylylene diisocyanate.

Among these, 2,4-tolylene diisocyanate, 2,6-tolylene diisocyanate, hydrogenated xylylene diisocyanate, isophorone diisocyanate, hydrogenated diphenylmethane diisocyanate, and the like are preferable. These diisocyanate compounds may be used either singly or in

PCT/NL99/00511

- 11 -

combinations of two or more.

WO 00/09620

Examples of the hydroxyl group-containing (meth)acrylate compound include 2-hydroxyethyl(meth)acrylate, 2hydroxypropyl (meth) acrylate, 2-hydroxybutyl (meth) acrylate, 2-hydroxy-3phenyloxypropyl (meth) acrylate, 1,4-butanediol mono(meth)acrylate, 2-hydroxyalkyl (meth)acryloyl phosphate, 4-hydroxycyclohexyl (meth) acrylate, 1,6-10 hexanediol mono(meth)acrylate, neopentyl glycol mono(meth)acrylate, trimethylolpropane di (meth) acrylate, trimethylolethane di (meth) acrylate, pentaerythritol tri(meth)acrylate, dipentaerythritol penta(meth)acrylate, and (meth)acrylates represented by 15 the following structural formula (2):

wherein R² represents a hydrogen atom or a methyl group and n denotes an integer from 1 to 15, and preferably from 1 to 4. Compounds obtained by the addition

20 reaction of a (meth)acrylic acid and a compound containing a glycidyl group such as alkyl glycidyl ether, allyl glycidyl ether, and glycidyl (meth)acrylate can also be given as examples of the (meth)acrylate compound. Among these compounds, 2
25 hydroxyethyl (meth)acrylate, 2hydroxypropyl (meth)acrylate, 2-

hydroxybutyl (meth) acrylate, and the like are

- 12 -

preferable.

There are no specific limitations to the process for synthesizing the urethane (meth)acrylate (A). For instance, synthesis of the urethane

- 5 (meth)acrylate (A) can be carried out according to the following processes (i) to (iii):
 - (i) A process of reacting the polyisocyanate compound
 - (b) with the hydroxyl group-containing (meth)acrylatecompound (c), and reacting the resulting product with
- 10 the polyol compound (a);
 - (ii) a process of reacting the polyol compound (a), the polyisocyanate compound (b), and the hydroxyl group-containing (meth)acrylate compound (c) altogether; and(iii) a process of reacting the polyol compound (a)
- with the polyisocyanate compound (b), and reacting the resulting product with the hydroxyl group-containing (meth)acrylate compound (c).

In the synthesis of the urethane

(meth)acrylate (A) used in the present invention, it is

preferable to use a urethanization catalyst such as
copper naphthanate, cobalt naphthanate, zinc
naphthanate, di-n-butyltin dilaurate, triethylamine,
1,4-diazabicyclo[2.2.2]octane, and 1,4-diaza-2methylbicyclo[2.2.2]octane in an amount from 0.01 to 1

part by weight for 100 parts by weight of the total
reaction components. The reaction is generally carried
out at a temperature of 0 to 90°C, and preferably 10 to
80°C.

The number average molecular weight of the urethane (meth)acrylate used in the present invention

- 13 -

is preferably from 400 to 20000, and more preferably from 600 to 10000.

To ensure superior adhesion to substrates and adequate viscosity, the urethane (meth)acrylate (A) is incorporated in the composition of the present invention in an amount preferably from 5 to 70 parts by weight for 100 parts by weight of the total amount of the components (A), (B), and (C).

As the (meth)acrylate compound having at

least one (meth)acryloyl group in the molecule which is

used as component (B) in the resin composition of the

present invention, either a monofunctional compound

having only one (meth)acryloyl group or a

polyfucntional compound having two or more

(meth)acryloyl groups, or combination of these at an

appropriate proportion, can be used.

Examples of the monofunctional compound include 2-hydroxyethyl(meth)acrylate, 2-hydroxypropyl(meth)acrylate, 2-

- hydroxybutyl (meth) acrylate, methyl (meth) acrylate, ethyl (meth) acrylate, propyl (meth) acrylate, isopropyl (meth) acrylate, butyl (meth) acrylate, amyl (meth) acrylate, isobutyl (meth) acrylate, t-butyl (meth) acrylate, pentyl (meth) acrylate, isoamyl
- (meth)acrylate, hexyl (meth)acrylate, heptyl
 (meth)acrylate, octyl (meth)acrylate, iso-octyl
 (meth)acrylate, 2-ethylhexyl (meth)acrylate, nonyl
 (meth)acrylate, decyl (meth)acrylate, isodecyl
 (meth)acrylate, undecyl (meth)acrylate, dodecyl
- 30 (meth)acrylate, lauryl (meth)acrylate, octadecyl

(meth)acrylate, stearyl (meth)acrylate,
tetrahydrofurfuryl (meth)acrylate, butoxyethyl
(meth)acrylate, ethoxydiethylene glycol (meth)acrylate,
benzyl (meth)acrylate, cyclohexyl (meth)acrylate,

- phenoxyethyl (meth)acrylate, polyethylene glycol
 mono(meth)acrylate, polypropylene glycol
 mono(meth)acrylate, methoxyethylene glycol
 mono(meth)acrylate, ethoxyethyl (meth)acrylate,
 ethoxyethoxyethyl (meth)acrylate, methoxypolyethylene
- glycol (meth)acrylate, methoxypolypropylene glycol (meth)acrylate, dicyclopentadienyl (meth)acrylate, dicyclopentanyl (meth)acrylate, dicyclopentenyl (meth)acrylate, tricyclodecanyl (meth)acrylate, bornyl (meth)acrylate, isobornyl (meth)acrylate, adamantyl
- (meth) acrylate, dimethylaminoethyl (meth) acrylate,
 diethylaminoethyl (meth) acrylate, 7-amino-3,7dimethyloctyl (meth) acrylate, (meth) acryloylmorpholine,
 2-(meth) acryloyloxyethylphthalic acid,
 - 2-(meth)acryloyloxyethylhexahydrophthalic acid,
- 20 2-(meth)acryloyloxypropylphthalic acid,
 - 2-(meth)acryloyloxypropyltetrahydrophthalic acid,
 - 2-(meth)acryloyloxypropylhexahydrophthalic acid,
 - 2-(meth)acryloyloxyethylsuccinic acid, trifluoroethyl (meth)acrylate, tetrafluoropropyl (meth)acrylate,
- hexafluoropropyl (meth)acrylate, octafluoropentyl (meth)acrylate, heptadecafluorodecyl (meth)acrylate, and compounds represented by the following formulas (3) to (5):

- 15 -

$$CH_{2} = C - C - O - (R^{3}O)_{p}$$

$$R^{4} O$$

$$R^{5}$$
(3)

wherein R³ represents an alkylene group or a hydroxyalkylene group having 2-6 carbon atoms, R⁴ represents a hydrogen atom or a methyl group, R⁵ represents a hydrogen atom or an alkyl group having 1-12 carbon atoms, and p denotes an integer from 0 to 20;

wherein R^6 represents a hydrogen atom or a methyl group, R^7 represents an alkylene group having 2-8 carbon atoms, and q denotes an integer from 0 to 8; and

$$CH_{2} = C - C - (OR^{9}C)_{r} - O - CH_{2} -$$

15

20

wherein R^8 represents a hydrogen atom or a methyl group, R^9 represents an alkylene group having 2-8 carbon atoms, r denotes an integer from 0 to 8, and R^{10} and R^{11} independently represent a hydrogen atom or an alkyl group having 1-6 carbon atoms.

These compounds are commercially available under the trade names, for example, Aronix M101, M102, M110, M111, M113, M114, M117, M120, M152, M154, M5300, M5400, M5500, M5600 (manufactured by Toagosei Co.,

Ltd.), KAYARAD TC-110S, R-128H, R629, R644

(manufactured by Nippon Kayaku Co., Ltd.), IPAA, AIB,
SBAA, TBA, IAAA, HEXA, CHA, NOAA, IOAA, INAA, LA, TDA,
MSAA, CAA, HDAA, LTA, STA, ISAA-1, ODAA, NDAA, IBXA,

- ADAA, TCDA, 2-MTA, DMA, Viscoat #150, #150D, #155, #158, #160, #190, #190D, #192, #193, #220, #320, #2311HP, #2000, #2100, #2150, #2180, MTG (manufactured by Osaka Organic Chemical Industry Co., Ltd.), NK Ester M-20G, M-40G, M-90G, M-230G, CB-1, SA, S, AMP-10G, AMP-
- 20G, AMP-60G, AMP-90G, A-SA, NLA (manufactured by Shin-Nakamura Chemical Co., Ltd.), ACMO (manufactured by Kojin Co., Ltd.), Light Acrylate IA-A, L-A, S-A, BO-A, EC-A, MTG-A, DPM-A, PO-A, P-200A, THF-A, IB-XA, HOA-MS, HOA-MPL, HOA-MPE, HOA-HH, IO-A, BZ-A, NP-EA, NP-10EA,
- HOB-A, FA-108, Epoxy Ester M-600A, (manufactured by Kyoeisha Chemical Co., Ltd.), and FA-511, FA-512A, FA-513A (manufactured by Hitachi Chemical Co., Ltd.).

Examples of a polyfunctional compound include ethylene glycol di(meth)acrylate, propylene glycol di(meth)acrylate, 1,4-butanediol di(meth)acrylate, 1,6-hexanediol di(meth)acrylate, 1,9-nonanediol di(meth)acrylate, diethylene glycol di(meth)acrylate, triethylene glycol di(meth)acrylate, tetraethylene glycol di(meth)acrylate, polyethylene

- glycol di(meth)acrylate, dipropylene glycol di(meth)acrylate, tripropylene glycol di(meth)acrylate, polypropylene glycol di(meth)acrylate, neopentyl glycol di(meth)acrylate, hydroxypivalic acid neopentyl glycol di(meth)acrylate, trimethylolpropane tri(meth)acrylate,
- 30 pentaerythritol tri(meth)acrylate,

pentaerythritol tetra(meth)acrylate,
ditrimethylolpropane tetra(meth)acrylate,
dipentaerythritol penta(meth)acrylate,
dipentaerythritol hexa(meth)acrylate,
trimethylolpropane trioxyethyl(meth)acrylate,
trimethylolpropane polyoxyethyl(meth)acrylate,
trimethylolpropane trioxypropyl(meth)acrylate,
trimethylolpropane polyoxyethyl(meth)acrylate,
trimethylolpropane polyoxyethyl(meth)acrylate,

tris(2-hydroxyethyl)isocyanurate tri(meth)acrylate, ethylene oxide addition bisphenol A di(meth)acrylate, ethylene oxide addition bisphenol F di(meth)acrylate, propylene oxide addition bisphenol A di(meth)acrylate, propylene oxide addition bisphenol F di(meth)acrylate,

tris(2-hydroxyethyl)isocyanurate di(meth)acrylate,

tricyclodecanedimethanol di(meth)acrylate, bisphenol A diepoxydi(meth)acrylate, and bisphenol F diepoxy di(meth)acrylate.

Examples of commercially available products of these compounds include SA-1002, SA-2006, SA-2007, 20 SA-4100, SA-5001, SA-6000, SA-7600, SA-8000, SA-9000 (manufactured by Mitsubishi Chemical Corp.), Viscoat #195, #195D, #214HP, #215, #215D, #230, #230D, #260, #295, #295D, #300, #310HP, #310HG, #312, #335HP, #335D, #360, #400, V#540, #700, GPT (manufactured by Osaka 25 Organic Chemical Industry Co., Ltd.), KAYARAD MANDA, R-526, NPGDA, PEG400DA, R-167, HX-220, HX-620, R-551, R-712, R-604, R-684, GPO-303, TMPTA, THE-330, TPA-320, TPA-330, PET-30, RP-1040, T-1420, DPHA, D-310, D-330, DPCA-20, DPCA-30, DPCA-60, DPCA-120 (manufactured by 30 Nippon Kayaku Co., Ltd.), Aronix M210, M208, M215.

10

WO 00/09620 PCT/NL99/00511

- 18 -

M220, M225, M233, M240, M245, M260, M270, M305, M309, M310, M315, M320, M350, M360, M400, M408, M450 (manufactured by Toagosei Co., Ltd.), SR-212, SR-213, SR-355 (manufactured by Sartomer), and SP-1506, SP-1507, SP-1509, SP-1519-1, SP-1563, SP-2500, VR60, VR77, VR90 (manufactured by Showa Highpolymer Co., Ltd.).

These (meth)acrylate compounds used as the component (B) is incorporated in the composition of the present invention preferably in an amount from 30 to 90 parts by weight for 100 parts by weight of the total amount of the components (A), (B), and (C).

Any known photo-polymerization initiator
may be used as component (C) in the composition of the
present invention without specific limitations. Given
15 as examples of the photo-polymerization initiator (C)
are 1-hydroxycyclohexyl phenyl ketone, 3methylacetophenone, 2,2-dimethoxy-2-phenylacetophenone,
xanthone, fluorenone, benzaldehyde, fluorene,
anthraquinone, triphenylamine, carbazole, 3-

- methylacetophenone, benzophenone, 4-chlorobenzophenone, 4,4'-dimethoxybenzophenone, 4,4'-diaminobenzophenone, benzoin ethyl ether, benzoin propyl ether, Michler's ketone, benzyl methyl ketal, 1-(4-isopropylphenyl)-2-hydroxy-2-methylpropan-1-one, 2-hydroxy-2-methyl-1-
- phenylpropan-1-one, 1-(4-dodecylphenyl)-2-hydroxy-2methylpropan-1-one,
 - 4-(2-hydroxyethoxy) phenyl-(2-hydroxy-2-propyl) ketone, 2-methyl-1-[4-(methylthio) phenyl]-2-morpholino-propan-1-one, 2,4,6-trimethylbenzoylphenyl phosphinate,
- 30 2,4,6-trimethylbenzoyldiphenylphosphine oxide, 2-

- 19 -

benzyl-2-dimethylamino-1-(4-morpholinophenyl)-butan-1one,

bis(2,6-dimethoxybenzoyl)-2,4,4-

trimethylpentylphosphine oxide, methylbenzoyl formate,

5 thioxanethone, diethylthioxanthone, 2isopropylthioxanthone,

2-chlorothioxanthone, and oligo[2-hydroxy-2-methyl-1-[4-(1-methylvinyl)phenyl]propanone].

These photo-polymerization initiators are 10 commercially available under the trade names, for example, IRGACURE 184, 261, 369, 500, 651, 907, CGI-403, 819, 1700, 1800, 1850 (manufactured by Ciba Specialty Chemicals Co., Ltd.), Lucirin TPO, TPO-L. LR8893 (manufactured by BASF), Darocur 953, 1116, 1173,

- 1664, 2273, 2959, ZL1 3331 (manufactured by Merck), 15 Ubecryl P36 (manufactured by UCB), VICURE55 (manufactured by Akzo), ESACURE KIP100F, KIP150 (manufactured by Lamberti), and KAYACURE ITX, QTX, DETX, BMS (manufactured by Nippon Kayaku Co., Ltd.). Of
- 20 these, IRGACURE 184, 369, 651, 907, Darocur 1173, CGI-1700, 1800, 1850, Lucirin TPO, and TPO-L are particularly preferred.

These photo-polymerization initiators may be used either individually or in combinations of two or more.

25

30

The component (C) is incorporated in the composition of the present invention in an amount usually from 0.1 to 20 parts by weight, preferably from 0.5 to 15 parts by weight, and particularly preferably from 1 to 10 parts by weight, for 100 parts by weight

- 20 -

of the total amount of the components (A), (B), and (C).

In addition to the above components (A),

(B), and (C), radically polymerizable compounds other

than compounds containing an acryl group may be

incorporated in the composition of the present

invention as a component (D). Given as examples of such

compounds are N-vinylpyrrolidone, N-vinylcaprolactam,

vinyl acetate, vinyl propionate, styrene,

5

divinylbenzene, and unsaturated polyesters. The
unsaturated polyesters are esters of a dicarboxylic
acid containing a radically polymerizable unsaturated
double bond and an alcohol. Examples of a dicarboxylic
acid containing a radically polymerizable unsaturated
double bond include maleic acid anhydride, itaconic

double bond include maleic acid anhydride, itaconic acid, and fumaric acid. Examples of an alcohol include monohydric alcohols such as methanol, ethanol, n-propyl alcohol, isopropyl alcohol, n-butyl alcohol, isobutyl alcohol, sec-butyl alcohol, tert-butyl alcohol, n-

hexanol, cyclohexanol, and 2-ethylhexyl alcohol;

(poly)ethylene glycols such as ethylene glycol,

diethylene glycol, triethylene glycol, and the like;

(poly)propylene glycols such as propylene glycol,

dipropylene glycol, and tripropylene glycol; dihydric

alcohols such as 1,6-hexanediol; and trihydric alcohols

such as glycerol and trimethylolpropane.

Furthermore, an epoxy resin, polyamide, polyamideimide, polyurethane, polybutadiene, chloroprene, polyether, polyester, pentadiene derivatives, SBS (styrene/butadiene/styrene block

- 21 -

copolymer), hydrogenated products of SBS, SIS (styrene/isoprene/styrene block copolymer), petroleum resin, xylene resin, ketone resin, fluorine-based oligomer, silicone-based oligomer, polysulfide-based oligomer, and the like may be incorporated in the composition of the present invention as other additives.

The composition of the present invention may further include various additives used for paints 10 other than the above additives, for example, antioxidants, UV absorbers, light stabilizers, aging preventives, silane coupling agents, antifoaming agents, leveling agents, antistatic agents, surfactants, preservatives, heat polymerization 15 inhibitors, plasticizers, and wettability improvers. Examples of antioxidants include Irganox 245, 259, 565, 1010, 1035, 1076, 1081, 1098, 1222, 1330 (manufactured) by Ciba Specialty Chemicals Co., Ltd.), and the like.

Examples of UV absorbers include

benzotriazole type and triazine type UV absorbers and 20 commercially available products such as Tinuvin P, 234, 320, 326, 327, 328, 213, 400 (manufactured by Ciba Specialty Chemicals Co., Ltd.), Sumisorb 110, 130, 140, 220, 250, 300, 320, 340, 350, 400 (manufactured by

25

Examples of light stabilizers include commercially available products such as Tinuvin 144, 292, 622LD (manufactured by Ciba Specialty Chemicals Co., Ltd.), Sanol LS440, LS770 (manufactured by Sankyo 30 Co., Ltd.), and Sumisorb TM-061 (manufactured by

Sumitomo Chemical Industries Co., Ltd.), and the like.

- 22 -

Sumitomo Chemical Industries Co., Ltd.).

Examples of aging preventives include phenol type, allylamine type, and ketone amine type aging preventives. Examples of commercially available products of these aging preventives include Antigene W, S, P, 3C, 6C, RD-G, FR, and AW (manufactured by Sumitomo Chemical Industries Co., Ltd.).

Examples of silane coupling agents include:

N-(2-aminoethyl)-3-aminopropylmethyldimethoxysilane,

10 N-(2-aminoethyl)-3-aminopropyltrimethoxysilane,

y-aminopropyltriethoxysilane,

γ-mercaptopropylmethylmonomethoxysilane,

γ-mercaptopropylmethyldimethoxysilane,

γ-mercaptopropyltrimethoxysilane,

15 γ-mercaptopropylmonoethoxysilane,

γ-mercaptopropyldiethoxysilane,

γ-mercaptopropyltriethoxysilane,

 β -mercaptoethylmonoethoxysilane,

 β -mercaptoethyltriethoxysilane,

20 β-mercaptoethyltriethoxysilane,

γ-glycidoxylpropyltrimethoxysilane,

y-glycidoxylpropylmethyldimethoxysilane,

2-(3,4-epoxycyclohexyl)ethyltrimethoxysilane,

γ-chloropropylmethyldimethoxysilane,

25 γ-chloropropyltrimethoxysilane, and γ-methacryloyloxypropyltrimethoxysilane. Examples of commercially available products of these compounds include Sila-Ace S310, S311, S320, S321, S330, S510, \$520, \$530, \$610, \$620, \$710, \$810 (manufactured by

Chisso Corp.), SH6020, SZ6023, SZ6030, SH6040, SH6076, SZ6083, AY43-062 (manufactured by Toray-Dow Corning Silicone Co., Ltd.), KBM403, KBM503, KBM602, KBM603, KBM803, KBE903 (manufactured by Shin-Etsu Silicone Co., Ltd.), and the like.

- 23 -

Examples of antifoaming agents include organic copolymers excluding silicon and fluorine which are commercially available under the trade names, for example, Flowlen AC-202, AC-300, AC-303, AC-326F, AC-

- 900, AC-1190, and AC-2000 (manufactured by Kyoeisha Yushi Co., Ltd.); silicon-containing antifoaming agents which are commercially available under the trade names, for example, Flowlen AC-901, AC-950, AC-1140, AO-3, AO-40H (manufactured by Kyoeisha Yushi Co., Ltd.), FS1265,
- SH200, SH5500, SC5540, SC5570, F-1, and SD5590

 (manufactured by Toray-Dow Corning Silicone Co., Ltd.);

 fluorine-containing antifoaming agents which are

 commercially available under the trade names, for

 example, MEGAFAC F-142D, F-144D, F-178K, F-179, F-815
- 20 (manufactured by Dainippon Ink and Chemicals, Inc.); and the like.

25

Examples of leveling agents include Polyflow No. 7, No. 38, No. 50E, S, No. 75, No. 77, No. 90, No. 95, No. 300, No. 460, ATF, KL-245 (manufactured by Kyoeisha Chemical Co., Ltd.), and the like.

The amount of these additives can be appropriately determined so that the objectives of the present invention are not adversely affected.

The amount of chlorine is defined as relative to the total composition, and more preferably

- 24 -

relative to total of components (A), (B) and (C). In the composition of the present invention some chlorine may be present, for instance, 0.1 ppm or more, but should not be more than 0.001 wt%, preferably not more than 0.0003 wt%, and even more preferably not more than 0.0005 wt%. Distortion on the surface of a metal film for optical disk substrate can be reduced using the resin composition with a small chlorine content. In addition, the moisture resistance and heat resistance of the optical disk substrate are improved. Reducing the chlorine content to less than 0.001 wt% can be achieved by using the raw materials such as components (A), (B), and (C), as well as other additives, having a chlorine content of less than 0.001 wt%. As some raw materials may have a chlorine content of more than 15 0.001 wt%, therefore, it may be necessary to wash these materials either before, during or after they are combined with or into the composition to lower or remove the chlorine. Preferred methods for reducing the 20 chlorine content in the composition of the present invention to less than 0.001 wt% include (1) a method of washing the raw materials, an intermediate containing two or more raw materials, or the composition prepared by mixing all the components with 25 purified water and (2) a method of treating one or more of these materials with an ion exchange material.

For example, the method for removing chlorine by washing preferably includes thoroughly mixing with purified water the treating material, i.e., (1) the raw materials, (2) the intermediate comprising

- 25 -

at least two raw material components, or (3) the composition containing all raw materials, with purified water to dissolve out chlorine into water, separating the water from the treating materials, and drying the treating material. Here, "purified water" means water with a sufficiently low content of chlorine. Preferably, purified water includes distilled water, ion exchanged water, and water prepared by filtering these waters through a suitable filter. For ease of 10 operation, an organic solvent may be added to the treating material prior to washing with water. Such an organic solvent is removed after washing. A hydrophobic solvent which can abundantly dissolve the raw materials, the intermediate comprising at least two raw 15 material components, or the composition containing all raw materials, but can only scarcely dissolve water, can be used in this washing operation.

Specific examples of such a solvent include aromatic hydrocarbons such as benzene, toluene, and 20 xylene; aliphatic hydrocarbons such as n-hexane, cyclohexane, and n-pentane; and ethers such as diethyl ether and isopropyl ether.

Suitable ion exchange materials include organic ion exchange materials and inorganic ion 25 exchange materials. A treatment by means of "contact" is a typical treatment with these ion exchange materials. A specific method of treatment comprises mixing the ion exchange material with the treating material, i.e., (1) the raw materials, (2) the 30 intermediate comprising at least two raw material

- 26 -

components, or (3) the composition containing all raw materials. After mixing, the ion exchange material is usually removed by precipitation in a sedimentation tank, forcedly separated by a centrifugal separator, or removed by filtration using a suitable filter. However, the ion exchange material need not be removed, if there is no adverse effect on the performance of the adhered disks. Moreover, it is possible to continuously remove chlorine by causing the treating materials to pass through a column packed with the ion exchange material.

The ion exchange material used in the present invention should be mutually insoluble with and easily separated from the resin composition, and is preferably in the form of particles. Suitable ion-

- 15 exchange resins include organic materials, and inorganic ion exchange materials. Examples of suitable inorganic materials having an ion exchange capabilities include activated carbon, zeolite, synthetic zeolite, silica gel, activated alumina, hydrotalcite,
- 20 hydrocalumite, acid clay, dolomite, zirconium hydroxide, titanium hydroxide, cerium hydroxide, bismuth hydroxide, manganese hydroxide, hydroxide lead phosphate, various hydrate oxide containing Mg, Zn, Ca, Mn, Fe, Co, Ni, Cu, Al, Si, or the like as a major
- 25 component, and inorganic ion exchange materials containing various metal phosphate as a major component. Preferred inorganic ion-exchange materials include hydrotalcite compounds are DHT-4A, KW-1000, KW-2000 (manufactured by Kyowa Chemical Industry Co.,
- . 30 Ltd.); as hydrocalumite compound, Solcat C

- 27 -

(manufactured by Nippon Chemical Industrial Co., Ltd.); as bismuth-type ion exchange materials, IXE-500, IXE-550, IXE-600, IXE-633, and IXE-680; as aluminum-type ion exchange materials, IXE-700 and IXE-702; as zirconium-type ion exchange material, IXE-800; as lead-based inorganic ion exchange material, IXE-1000; as calcium-type ion exchange material, IXE-1100; and as antimony-type ion exchange material, IXE-1320 (all manufactured by Toagosei Co., Ltd.).

The viscosity of the composition of the present invention is preferably from 10 to 10000 mPa.s, more preferably from 50 to 5000 mPa.s, and even more preferably from 100 to 2000 mPa.s.

These components are formulated so that the
glass transition temperature of the resulting cured
product is from -50 to 200°C, preferably from -20 to
150°C, and more preferably from 0 to 100°C. If the glass
transition temperature is too low, the cured products
may soften when heated in summer or in a closed and
sunny room, thereby resulting in decrease in adhesion
strength. This may cause the adhered members to peel
off or slip out. On the contrary, if the glass
transition temperature is too high, adhesion may be
insufficient or the coated members break when dropped
or bent.

The glass transition temperature described here is defined as the temperature at which a loss tangent (tan δ) reaches a peak when measured by a dynamic viscoelasticity measuring device at an oscillation frequency of 10 Hz.

- 28 -

The composition of the present invention can be cured by exposure to ultraviolet radiation, visible rays, electron beams, or the like in the same manner as in the case of using conventional

5 photocurable resin compositions. The composition of the present invention can be easily cured by filling the composition between the members to be coated so that the thickness of the adhesive layer is from 10 to 100 mm and irradiating the adhesive layer at a dose of 50 to 2000 J/cm² using a metal halide lamp, whereby the members can be glued to each other.

Further, the cured products made from the composition of the present invention are desired to have excellent transparency. For example, it is 15 preferable that the cured products with a thickness of 60 mm have a light transmittance of 90% or more at a wave length of 400 to 700 nm. If the light transmittance is less than 90%, not only is the appearance of the optical disks impaired, but also the 20 light for reading the information recorded on the disk is reduced by the adhesive layer, thereby making it difficult to read the information. Therefore, in preparing the composition of the present invention, each component should be blended so that the light 25 transmittance of the resulting cured product satisfy this requirement.

In addition, it is desirable to blend each component so that the refractive index of the photocured product made from the composition of the present invention is in the range from 1.51 to 1.70. If

- 29 -

the refractive index is out of this range, reading of the information recorded on the disk may be difficult.

The composition of the present invention exhibits excellent adhesion to plastics such as polycarbonate (PC) and poly(methylmethacrylate) (PMMA), metals such as gold and aluminum, and inorganic materials such as glass. Therefore, the composition of the present invention is suitable as an adhesive for various elements of an information disk in particular those employed in optical disks.

10

The resin composition of the present invention is useful as an adhesive for information disks made by adhering two sheets of substrate, particularly for adhering a layer comprising information (or material suitable for recording 15 information) which may consist, for example, of a light reflection layer made of a thin metal film provided on a resin substrate, to a second substrate. Often, the second substrate may similarly comprise information (or 20 material suitable for recording information) consisting of a light reflection layer made of a thin metal film provided on a resin substrate, or another element not comprising an information storage/recording components. Preferred examples of an information disk comprising 25 multiple elements include DVD, DVD-R, DVD-ROM, and the like.

If the chlorine content relative to the total composition of the present invention exceeds 0.001 wt%, corrosion or deterioration of the thin metal film on the information recording layer will be

20

conspicuous due to moisture invading from outside into the cured adhesive layer or into the interface of the cured adhesive and metal film.

Preferred embodiments of the adhesive for optical disks of the present invention described above in detail are as follows:

- 1. The composition, wherein the urethane (meth)acrylate for the component (A) has a number average molecular weight from 600 to 10,000.
- 2. The composition, wherein the component (A) is incorporated in an amount from 5 to 70 parts by weight of 100 parts by weight of the total amount of the components (A), (B), and (C).
- The composition, wherein the component (B) is incorporated in an amount from 30 to 90 parts by weight of 100 parts by weight of the total amount of the components (A), (B), and (C).
 - 4. The composition, wherein the component (C) is incorporated in an amount from 1 to 10 parts by weight of 100 parts by weight of the total amount of the components (A), (B), and (C).
 - 5. The composition having a chlorine content of not more than 0.0005% by weight.
- 6. The composition having a viscosity from 100 to 2000 mPa.s at 25°C.
 - 7. The composition, of which the cured product obtained by irradiation with UV light has a glass transition temperature from 0 to 100°C.
- 8. The composition, of which the cured product obtained 30 by irradiation with UV light has a refractive index

- 31 -

from 1.51 to 1.70 at 25°C.

Examples

The present invention will be explained in more detail by way of examples, which are not intended to be limiting of the present invention.

Synthesis of urethane acrylate for component (A)

Synthesis Example 1

130.3 g of isophorone diisocyanate, 0.3 g of 3,5-di-t-butyl-4-hydroxytoluene, and 1.3 g of di-n-10 butyltin dilaurate were placed in a separable flask (1 liter) equipped with a stirrer and a thermometer. The mixture was stirred and cooled to 10°C in a cold water bath in dry air. 109 g of 2-hydroxyethyl acrylate was gradually added to the mixture and reacted for one hour 15 while maintaining the temperature at 10 to 35°C. Then, 600 g of polytetramethylene glycol having a hydroxyl value of 54.9 mg KOH/g was added and the resulting mixture was stirred at 50 to 70°C for 5 hours to react, 20 thus obtaining urethane acrylate (A1) having a number average molecular weight of 2710.

200 g of the urethane acrylate (A1) was dissolved in 400 g of toluene. After the addition of 1000 g of ion exchanged water, the solution was thoroughly stirred and then allowed to stand to separate the water layer from the organic layer. After the addition of 1000 g of ion exchange water to the resulting organic layer, the mixture was stirred and allowed to stand, followed by separation of the water layer. The organic layer thus obtained was subjected to

- 32 -

a rotary evaporator to evaporate toluene and water, thus obtaining purified urethane acrylate (A2).

Synthesis Example 2

5 175.8 g of 1,3-

bis(isocyanatemethyl)cyclohexane (Takenate 600, manufactured by TAKEDA CHEMICAL INDUSTRIES Co., Ltd.), 0.3 g of 3,5-di-t-butyl-4-hydroxytoluene, and 505.6 g of polycarbonate diol having a hydroxyl value of 224 mg KOH/g (PLACCEL CD205, manufactured by Daicel Chemical Industries, Ltd.) were placed in a separable flask (1 liter) equipped with a stirrer and a thermometer. The mixture was stirred and cooled to 10°C in a cold water bath in dry air. 1.3 g of di-n-butyltin dilaurate was 15 added and the mixture was stirred for one hour while cooling over a cold water bath to maintain the temperature at 10 to 35°C. Then, 17.2 g of 2hydroxyethyl acrylate was added and the resulting mixture was stirred at 50 to 70°C for 5 hours to continue the reaction, thus obtaining urethane acrylate (A3) having a number average molecular weight of 1130.

The urethane acrylate (A3) was washed with ion exchanged water in the same manner as in Synthesis Example 1 to obtain urethane acrylate (A4).

25

30

20

10

Examples and Comparative Examples

Preparation of photocurable resin compositions

The components shown in Table 1 were placed in a reaction vessel equipped with a stirrer and stirred to prepare the compositions of Examples 1-5 and

- 33 -

Comparative Examples 1-4. The amount of each component is indicated by parts by weight in Table 1. After preparation, the compositions of Example 1 and Example 3 were passed through a glass tube with a diameter of 5 cm packed with granules of a bismuth-type ion exchange material (IXE-500G, manufactured by Toagosei Co., Ltd.) to a depth of 50 cm and maintained at 50°C, thereby removing chloride ion. This treatment for removing chloride ion is hereinafter called "column treatment".

10 The components shown in Table 1 are as follows.

Component (B)

B1: Phenoxyethyl acrylate

B2: 1,6-Hexanediol diacrylate

15 B3: Lauryl acrylate

B4: 2-Hydroxy-3-phenyloxypropyl acrylate

B5: Acryloylmorpholine

B6: Tetrahydrofurfuryl acrylate

20 Component (C)

C1: 1-Hydroxycyclohexyl phenyl ketone

C2: 2-Methyl-1-[4-(methylthio)phenyl]-2-morpholinopropan-1-on

C3: Oligo[2-hydroxy-2-methyl-1-[4-(1-

25 methylvinyl)phenyl] propan-1-on] (Esacure KIP150, manufactured by Lamberti)

C4: 2,2-Dimethoxy-1,2-diphenylethan-1-on

Component (D) (other component)

30 D1: Sodium chloride

The chlorine content for the compositions (Examples 1-5 and Comparative Examples 1-4) thus prepared were measured as follows.

5 4 g of each composition shown in Table 1 and 80 g ion exchanged water were sealed in a Teflon container and heated in a pressure cooker for 24 hours at 120°C to hydrolyze hydrolyzable chlorine compounds into chloride ion. The chloride ion was dissolved in 10 ion exchanged water and quantitatively analyzed by ion chromatography using DIONEX 2010i manufactured by DIONEX Co. Ion Pac AS12A was used as a separation column and an aqueous solution of Na₂CO₃ (0.0027 mol/liter) and NaHCO₃ (0.0003 mol/liter) used as an 15 eluate at a flow rate of 1.1 ml/minute.

Viscosity of the compositions prepared above, refractive index and glass-transition temperature of the cured products made from the compositions, and moisture resistance and heat resistance of disks prepared by using these compositions as adhesives were evaluated according to the following method.

(1) Viscosity

20

25 Viscosity was measured at 25°C using a Brookfield type viscometer (BM-type, Rotor No. 2) manufactured by Tokyo Keiki Co., Ltd.

(2) Refractive index

30 The composition was applied on a flat glass

PCT/NL99/00511 WO 00/09620

- 35 -

plate using an applicator bar and cured by irradiating with ultraviolet radiation at a dose of 1 J/cm2 using a conveyer-type ultraviolet radiation apparatus with a metal halide lamp as a light source (manufactured by the Eye Graphics Co., Ltd.). The cured product was peeled off from the glass plate to obtain a cure film with a thickness 0.2 mm. Refractive index of the cure film was measured at 25°C using an Abbe's refractometer manufactured by ATAGO Co., Ltd.

10

(3) Glass-transition temperature

Glass-transition temperature of the cure film prepared in (2) above was measured using an enforced resonance frequency-type dynamic

15 viscoelasticity measurement appratus manufactured by Orientech Co., Ltd. A temperature when the loss tangent $(\tan\delta)$ was a peak at an oscillation frequency of 10 Hz was taken as the glass-transition temperature of the cured product.

20

30

(4) Moisture resistance and heat resistance of disks A polycarbonate substrate with a thickness of 0.6 mm and another substrate prepared by sputtering an aluminum film by vapor deposition to a thickness of 25 200 angstrom on one side of the same polycarbonate substrate were used for the test. The compositions prepared in the above Examples and Comparative Examples were dropped onto the aluminum surface of the latter substrate, i.e. the substrate with an aluminum film sputtered by vapor deposition. The polycarbonate

PCT/NL99/00511 WO 00/09620

- 36 -

substrate was layered over the aluminum surface onto which the composition was dropped. The substrates were rotated by a spin coater to cause the composition to permeate into the both substrates. Light was irradiated 5 at a dose of 300 mJ/cm² through the polycarbonate substrate to cure the composition. The adhered substrate thus prepared was allowed to stand for 200 hours in a thermo-hygrostat at 80°C, 95%RH, to observe the outward appearance both by the naked eyes and microscope. The moisture resistance and heat resistance was rated as "O" for the composition exhibiting no uniform corrosion, pitting corrosion, intergranular corrosion, or corrosion breaking on aluminum surface. Otherwise, the moisture resistance and heat resistance was rated as "X".

10

															_	
	4				20				15	10	20			Э	7	15.
Example	m				20				15	10	20			٣	7	10
Comparative Example	2			20					15 ,	10	20			m	7	
O)	1	40				15	30	10				3	2			
	2				20				15	10	20			3	2	2
	4				20				15	10	20			m	7	
Example	æ			20					15	10	20			æ	2	
	2		40			15	30	10				3	7			
	1	40				15	30	10				3	7			
		A1	A2	A3	A4	B1	B2	. B3	B4	BS	B6	C1	C	ຍ	C4	D1 (10 ⁻⁴)

TABLE 1

			Example			Col	Comparative Example	Example	
	1	2	3	4	5	1	2	٣	4
Column	Treated	None	Treated	None	None	None	None	None	None
treatment									
Viscosity	400	400	1500	1500	1500	400	1500	1500	1500
(mPa.s)									
Chlorine	9.0	1.3	0.7	5.4	9.1	12.6	20.4	11.8	14.2
content									
(10 ⁻⁴ wt%)									
Refractive	1.51	1.51	1.52	1.52	1.52	1.51	1.52	1.52	1.52
index									
(n _D ²⁵)									
Tg (°C)	45	45	63	63	63	45	63	63	63
Moisture	0	0	0	0	0	×	×	×	×
and heat									
resistance									
							THE REAL PROPERTY AND PERSONS ASSESSMENTS.	The second secon	

- 39 -

The viscosity of the resin compositions of Examples and Comparative Example, all comprising the components (A), (B), and (C) of the present invention, at 25°C was between 400 and 1500 mP·s. The glass transition temperature and refractive index at 25°C of the cured products obtained from these composition by irradiation of ultraviolet radiation was respectively from 45 to 63°C and from 1.51 to 1.52, indicating that all compositions satisfy the standard required for the 10 adhesive used for preparing information record disks. However, in the evaluation of the moisture and resistance indicating corrosivity of aluminum in the disks fabricated by adhering two sheet of substrates, the compositions of the Examples with a chlorine 15 content of less than 0.001 wt% exhibited no corrosion, whereas the disks fabricated using the compositions of the Comparative Examples containing more than 0.001 wt% of chlorine produced corrosion, failing to pass through the corrosion test.

The photocurable resin composition of the present invention comprises specific components and has a chlorine content of not more than 0.001 wt%. Optical disks fabricated by using the photocurable resin composition of the present invention as an adhesive exhibit superior heat resistance and moisture resistance.

- 40 -

CLAIMS

- A photocurable resin composition, comprising:
 - (A) an urethane (meth) acrylate compound,
- (B) a (meth)acrylate compound having at least one (meth)acryloyl group in the molecule, and(C) a photo-initiator,
 - wherein the content of chlorine relative to the total composition is not more than 0.001 wt%.
- A process for preparing the photocurable resin composition according to claim 1, comprising: washing components (A), (B), and/or (C).
 - 3. The process according to claim 2, wherein the washing includes contacting one or more of the components with purified water.

15

- 4. The process according to any one of claim 2-3, wherein the washing includes treating one or more of the components with an ion exchangeable substance.
- 20 5. An information disk comprising two elements affixed to each other with the photocurable resin composition according to claim 1.
 - 6. The information disk according to claim 5 wherein at least one of the elements comprises information.
 - 7. The information disk according to any one of claims 5-6, wherein the information disk comprises a mutli-layer structure.
- 8. The information disk according to any one of claims 5-7, wherein the information disk

- 41 -

comprises a plurality of layers comprising information.

9. The information disk according to any one of claims 5-8, wherein the information disk is a digital optical disk.

5

10. The information disk according to any one of claims 5-9, wherein the information disk is a DVD.





INTERNATIONAL SEARCH REPORT

PC., NL 99/00511

ž

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C09J4/06 G11B7/24 C08F290/14 C08F290/06 C08G18/67

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT					
Category :	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.			
P,X	WO 98 36325 A (TAKAHASHI TOSHIHIKO ;FURUTA RYOJI (JP); JSR CORP (JP); TAKASI HIDE) 20 August 1998 (1998-08-20) examples 1-5,COMP,1 claims	1,5-10			
P,X	EP 0 889 465 A (SUMITOMO CHEMICAL CO) 7 January 1999 (1999-01-07) abstract examples 1-4,COMP,2,3,5,6	1,5-10			
Α	EP 0 835 917 A (NIPPON KAYAKU KK) 15 April 1998 (1998-04-15) examples claims/	1-10			

Patent family members are listed in annex.
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the
invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled
in the art. "&" document member of the same patent family
Date of mailing of the international search report
24/11/1999
Authorized officer Pollio, M

Form PCT. ISA 210 (second sheet) (July 1992)



INTERNATIONAL SEARCH REPORT



International Application No PC / NL 99/00511

		PC./NL 99/00511
	ation) DOCUMENTS CONSIDERED TO BE RELEVANT	
Calegory *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 014, no. 337 (P-1079), 20 July 1990 (1990-07-20) & JP 02 116034 A (MITSUBISHI PLASTICS IND LTD), 27 April 1990 (1990-04-27) abstract	1-10
A	PATENT ABSTRACTS OF JAPAN vol. 018, no. 353 (P-1764), 4 July 1994 (1994-07-04) & JP 06 089462 A (THREE BOND CO LTD), 29 March 1994 (1994-03-29) cited in the application abstract	1-10
A	PATENT ABSTRACTS OF JAPAN vol. 010, no. 341 (P-517), 18 November 1986 (1986-11-18) & JP 61 142545 A (RICOH CO LTD), 30 June 1986 (1986-06-30) cited in the application abstract	1-10
	-	
		-